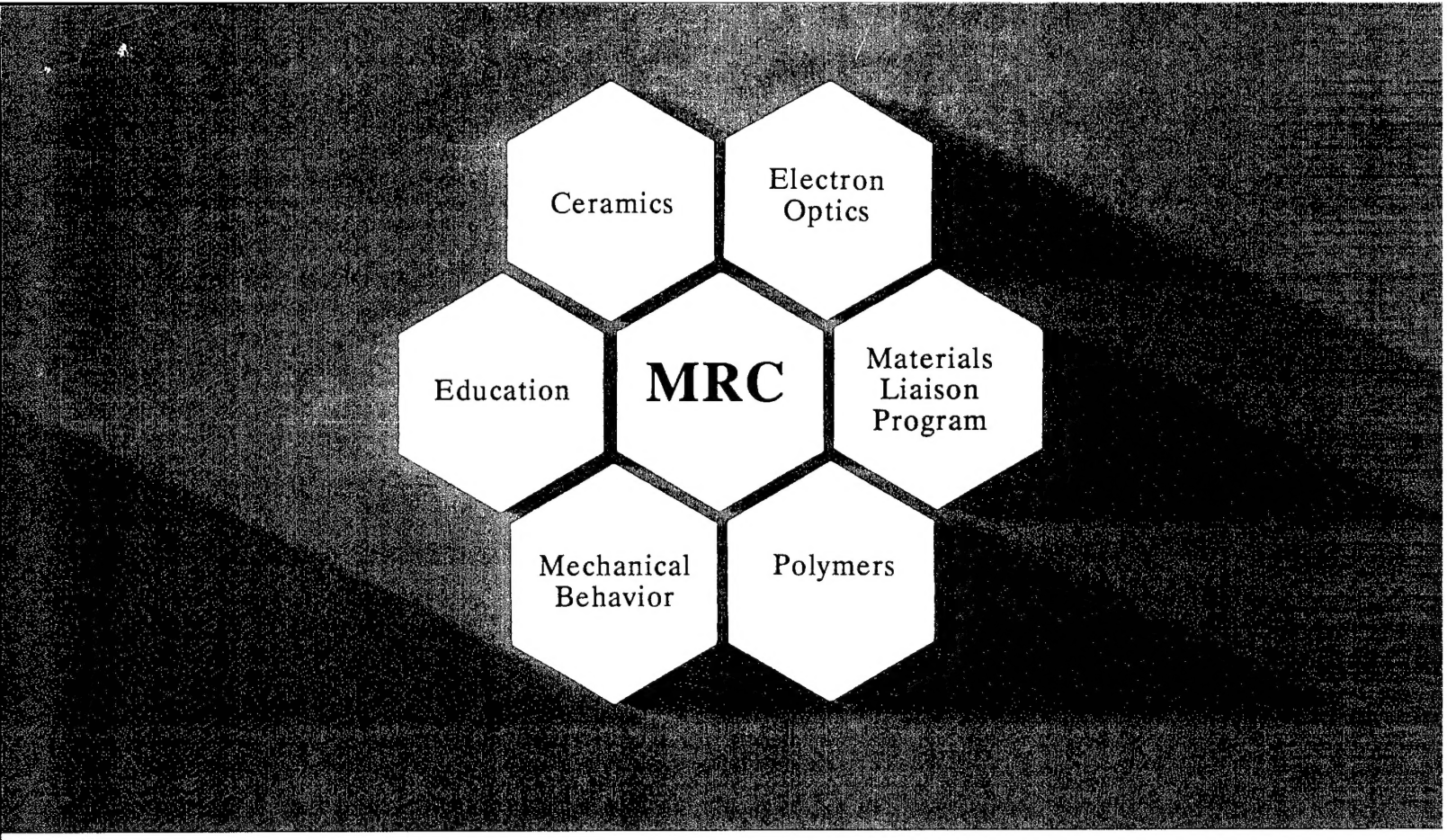

FINAL REPORT

A FOCUSED-ION BEAM (FIB) NANO-FABRICATION AND CHARACTERIZATION FACILITY

Professor M.P. Harmer (P.I)

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A Focused-Ion-Beam (FIB) Nano-Fabrication and Characterization Facility.

Project director Martin Harmer, Department of Materials Science and Engineering, Lehigh University.

Final report on the acquisition of a dual-beam focused ion beam system (henceforth referred to as the FIB).

This has been a most successful project. The FIB has been purchased, installed and is now the most productive instrument in the Microscopy Center at Lehigh. Substantial research has been accomplished by the group of the principal investigator, but much research has been done by many other groups as well. Much of the work has involved the preparation of samples for examination in the transmission electron microscope (TEM) and scanning electron microscope (SEM), but - as anticipated in the proposal - several experiments have already used the FIB for novel methods of nanofabrication of devices.

Research on novel piezoelectric devices

The materials studied are two-phase structures of lead-magnesium-niobate/lead-titanate (PMN-PT) with a second phase of lead oxide (PbO). The FIB is being used to create new innovative piezoelectric devices from these lead-based ceramics. One important application of these devices is as transducers that can be used for medical ultrasound components. Revolutionary performance may be obtained in these devices if each element (meaning each individual component of the device) is produced on the micron scale. Therefore the FIB was used to "mill out" trenches between individual elements on a scale that could not be accomplished in any other manner. The trenches are 5 μm wide, while the elements are 8 μm wide as shown in Figure 1. Theoretically this structure should lead to the best performing transducer ever built but the characterization of the piezoelectric properties needs to be completed to verify this claim. These preliminary experiments are most promising as an indication of the power of the technique to develop nanoscale devices in general.

The FIB was also used to prepare samples for TEM studies on the same samples: PMN-PT with PbO. Specific regions of bulk specimens were identified in the SEM and isolated with the FIB, as shown in Figure 2(a). The TEM micrographs in Figure 2(b) and 2(c) were obtained from the exact sample shown in Figure 2(a) after extraction. The samples were further characterized using analytical electron microscopy (AEM) in a high-resolution scanning transmission electron microscope (STEM). Absorption corrected chemical maps, such as that shown in Figure 2(d), revealed apparent changes in chemistry that took place during the firing cycle of the PMN-PT ceramics. These maps

were subsequently used to quantify the chemistry of the grains and the liquid phase and verify that the liquid phase chemistry has a profound effect on grain growth in this system.

Cathodoluminescence on biased structures (Group of Slade Cargill)

The FIB has been used to deposit electrodes and contacts onto structures of Gallium Nitride and related materials. In this way the effect on luminescence, of varying the bias on the device, can be measured. See figure 3.

Fabrication of photonic crystals (Group of Jean Toulouse, Department of Physics)

This group has begun fabricating photonic crystals with the FIB. The particular excitement of the work of the group of Dr. Toulouse is the way in which photonic crystals can be fabricated with defects in the periodic array.

Lucent Technologies have been doing some similar work related to fabricating periodic structures for use as optical gratings. (This is one of several projects by Lucent details of the others which are proprietary can not be given.)

Preparation of samples for TEM and SEM observation.

Several groups from universities and from industry have used the FIB for electron microscope sample preparation.

These include:
Professor Vinci (Lehigh) Copper/aluminum grain size in strained thin films.
Professor Cargill (Lehigh) Cross-sections of devices for SEM observation.
Professor Mohney (Penn State) Professor Mohney came to Lehigh on sabbatical especially to use the FIB for the examination of metallization contacts to optical semiconductors.
Professor Luzzi (U Pennsylvania) TEM sample preparation
Professor Levy (Michigan State) TEM sample preparation of irradiated oxides.
Professor Williams (Lehigh) TEM sample preparation for the study of grain-boundary segregation.
Professor DuPont (Lehigh) Making TEM samples for microanalysis from regions at specific positions with respect to a weld.
Industrial users include: Lucent; Scientific Design; Air Products; and ESC, Inc.

Financial

The funds provided under the grant were very much less than those requested. Fortunately we were able to negotiate with the vendor an extraordinary deal that allowed us to purchase the Dual-Beam FIB with the funds available. The importance of the Dual-Beam instrument is that the SEM incorporated into the FIB can be used to image the sample and track the progress of the FIB machining without eroding the sample. The

instrument was delivered in a very stripped-down form, as a result of the limited funding and there is a need to equip it with several accessories in order to obtain the maximum benefit for this program and in general. The addition of these items is the subject of a separate proposal.

Summary and Conclusions

The purchase of the FIB has been one of the most productive pieces of research funding that we have seen. The instrument was delivered quickly, has functioned well and contributed to many pieces of important research.

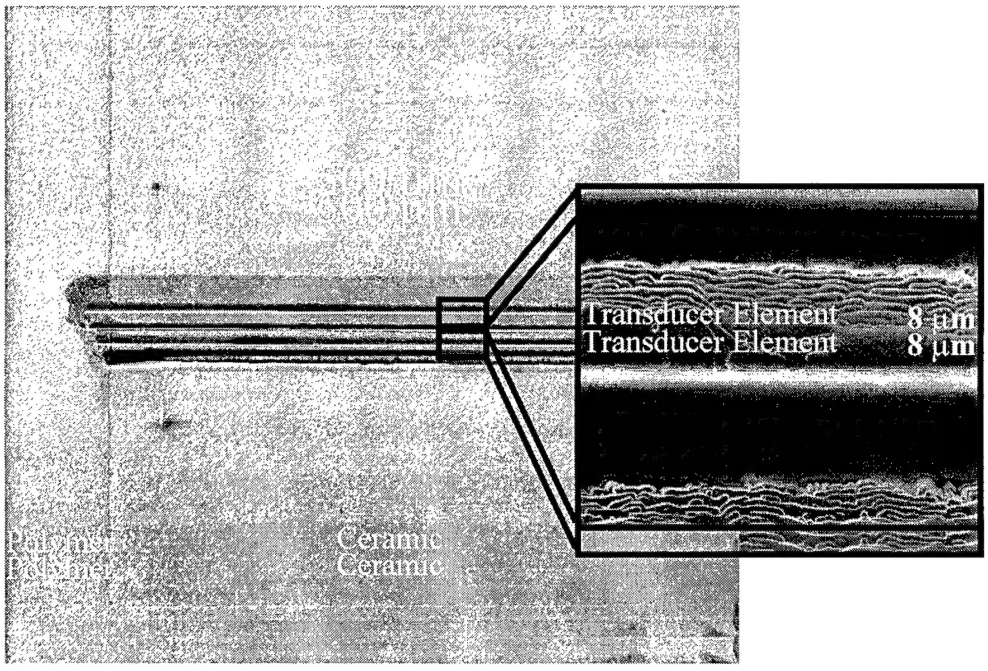

Figure 1. SEM image of a transducer being created using the FIB instrument.

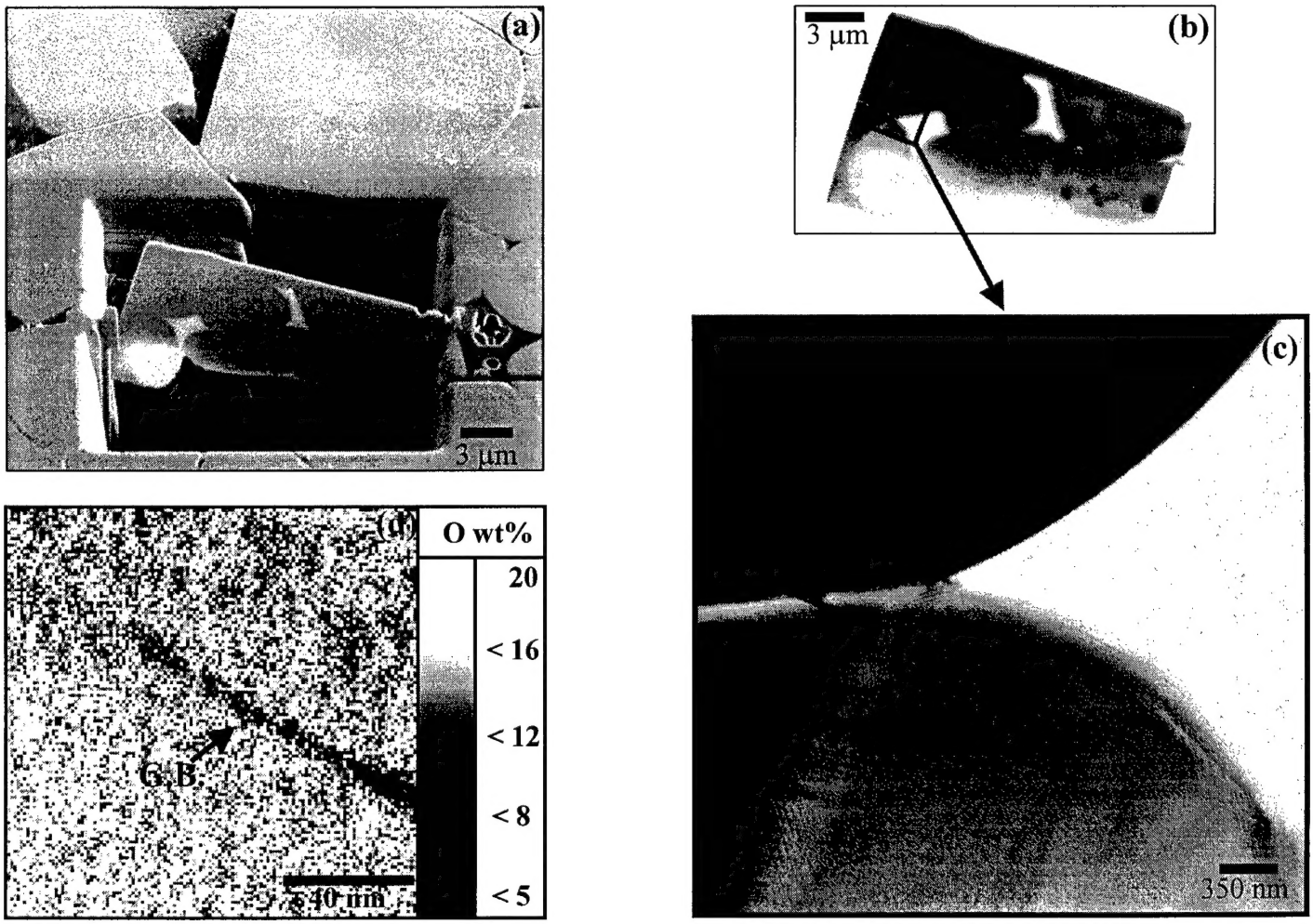

Figure 2. (a) Secondary electron image of a TEM sample prepared using a dual-beam FIB. (Note the bright areas are indications of electron transmission in SEM mode). **(b)** Bright field transmission electron micrograph of a FIB- prepared sample; **(c)** high magnification TEM image of a grain boundary containing a liquid phase that is adjacent to a liquid pocket. **(d)** X-ray chemical map of the weight fraction of oxygen at the grain boundary.

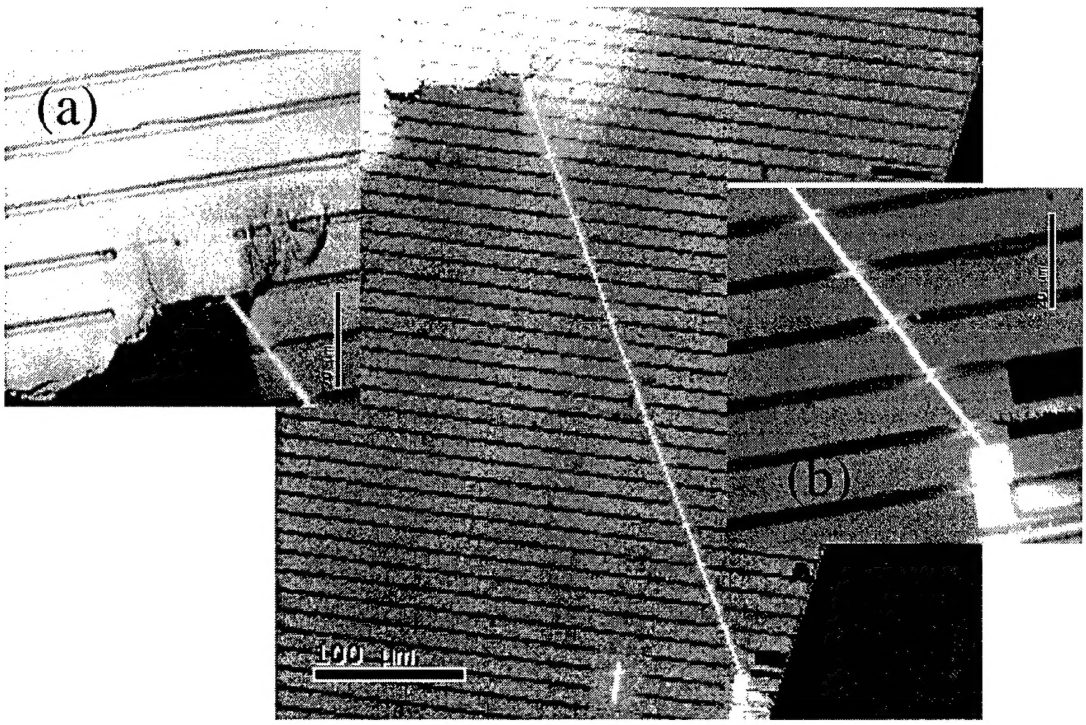

Figure 3 Backscattered electron images in the SEM show Pt contacts drawn with the FIB. Inset (a) shows the contact to a larger gold pad, and inset (b) shows a contact to a smaller platinum pad. The line is approximately 500 microns long.